REPORT DOCUMENTATION PAGE

Form Approved OMB NO. 0704-0188

1. REPORT DATE (DD-MM-YYYY)	2. REPORT TYPE	3. DATES COVERED (From - To)
25-07-2018	Final Report	28-Apr-2017 - 27-Apr-2018

4. TITLE AND SUBTITLE
Final Report: Equipment for the Optical Characterization of AlGaN Nanowire Heterostructures for Deep Ultraviolet Optoelectronics

5a. CONTRACT NUMBER
W911NF-17-1-0235

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER
611103

6. AUTHORS

5d. PROJECT NUMBER

5e. TASK NUMBER

5f. WORK UNIT NUMBER

7. PERFORMING ORGANIZATION NAMES AND ADDRESSES
University of Michigan - Ann Arbor
3003 South State Street

Ann Arbor, MI 48109 -1274

8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS (ES)
U.S. Army Research Office
P.O. Box 12211
Research Triangle Park, NC 27709-2211

10. SPONSOR/MONITOR'S ACRONYM(S)
ARO

11. SPONSOR/MONITOR'S REPORT NUMBER(S)
70076-EL-RIP.2

12. DISTRIBUTION AVAILIBILITY STATEMENT
Approved for public release; distribution is unlimited.

13. SUPPLEMENTARY NOTES
The views, opinions and/or findings contained in this report are those of the author(s) and should not contrued as an official Department of the Army position, policy or decision, unless so designated by other documentation.

14. ABSTRACT

15. SUBJECT TERMS

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	15. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
a. REPORT	b. ABSTRACT	c. THIS PAGE			Zetian Mi
UU	UU	UU	UU		19b. TELEPHONE NUMBER 734-764-3963

RPPR Final Report
as of 31-Jul-2018

Agency Code:

Proposal Number: 70076ELRIP **Agreement Number: W911NF-17-1-0235**

INVESTIGATOR(S):

Name: Zetian Mi
Email: ztmi@umich.edu
Phone Number: 7347643963
Principal: Y

Organization: **University of Michigan - Ann Arbor**
Address: 3003 South State Street, Ann Arbor, MI 481091274
Country: USA
DUNS Number: 073133571 EIN: 386006309

Report Date: 27-Jul-2018 Date Received: 25-Jul-2018
Final Report for Period Beginning 28-Apr-2017 and Ending 27-Apr-2018
Title: Equipment for the Optical Characterization of AlGaN Nanowire Heterostructures for Deep Ultraviolet Optoelectronics
Begin Performance Period: 28-Apr-2017 **End Performance Period:** 27-Apr-2018
Report Term: 0-Other
Submitted By: Zetian Mi Email: ztmi@umich.edu
Phone: (734) 764-3963
Distribution Statement: 1-Approved for public release; distribution is unlimited.

STEM Degrees: 0 **STEM Participants:** 0

Major Goals: In this project, we propose to investigate the epitaxy and structural and optical properties of Al-rich AlGaN nanowire heterostructures. The nanowires are grown using the special technique of selective area epitaxy to achieve a precise control of their size, spacing, and surface morphology, and the formation of quantum dots is incorporated to achieve high efficiency, tunable emission in the deep UV spectral range. We request a deep UV photoluminescence system to characterize the optical properties of AlGaN nanowire heterostructures. The system includes pump lasers, high resolution spectrometer, deep UV CCD, cryostat, mirrors, lenses, and other related optical components. Such a system is essential required for the research and development of AlGaN nanowire light sources, including LEDs and electrically pumped lasers operating in the UV-C band.

Accomplishments: We have built a deep ultraviolet photoluminescence system to characterize the optical properties of AlGaN nanowire heterostructures. With the use of this system, we have investigated the optical properties of AlGaN nanowire heterostructures emitting in the UV-B and UV-C bands. Major accomplishments are detailed in the attached report.

Training Opportunities: PhD students David Laleyan and Xianhe Liu have been trained in the optical characterization of wide bandgap AlGaN nanowire nanostructures. Master student Chan Ho Soh has also been trained on the operation of deep UV photoluminescence measurement system.

Results Dissemination: Conference presentations:

Invited: Z. Mi, “AlGaN Nanowire Deep Ultraviolet Photonics,” 233rd ECS Meeting, Seattle, WA, May 13-17, 2018.

Keynote: Z. Mi, “AlGaN nanowire LEDs and laser diodes operating in the UV-C band,” International Workshop on UV Materials and Devices (IWUMD), Fukuoka, Japan, Nov. 14-18, 2017.

Invited: Z. Mi, “AlGaN nanowire deep ultraviolet opotelectronics,” 33rd North American Conference on Molecular Beam Epitaxy, Galveston Island, TX, Oct. 15-18, 2017.

X. Liu, S. Zhao, B. H. Le, and Z. Mi, “Deep ultraviolet AlGaN Nanowire Light Emitting Diodes by Selective Area Epitaxial Growth,” International Symposium on Semiconductor Light Emitting Devices, Banff, Canada, Oct. 8-12, 2017.

D. A. Laleyan, S. Zhao, S. Y. Woo, H. N. Tran, H. B. Le, T. Szkopek, H. Guo, G. A. Botton and Z. Mi, “AlN/h-BN Nanowire Heterostructures for Deep Ultraviolet Photonics,” International Symposium on Semiconductor Light Emitting Devices, Banff, Canada, Oct. 8-12, 2017.

S. Zhao, S. Sadaf, X. Liu, and Z. Mi, “AlGaN Nanowire Tunnel Junction Light Emitting Diodes and Lasers,” International Symposium on Semiconductor Light Emitting Devices, Banff, Canada, Oct. 8-12, 2017.

Honors and Awards: Zetian Mi has been elected Fellow of SPIE and Fellow of OSA.

David Laleyan has received Best Student Poster Award at the 11th International Symposium on Semiconductor Light Emitting Devices.

Xianhe Liu has received Best Student Poster Award at the 11th International Symposium on Semiconductor Light Emitting Devices.

Protocol Activity Status:

Technology Transfer: Nothing to Report

PARTICIPANTS:

Participant Type: PD/PI
Participant: Zetian Mi
Person Months Worked: 1.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Graduate Student (research assistant)
Participant: David Laleyan
Person Months Worked: 3.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Graduate Student (research assistant)
Participant: David Laleyan
Person Months Worked: 3.00 **Funding Support:**

Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Graduate Student (research assistant)
Participant: Xianhe Liu
Person Months Worked: 2.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Project Report

1. Introduction and Project Objective

In the past decades, tremendous progress has been made in the research and development of AlGaN quantum well UV LEDs and lasers. However, compared to the extremely high performance GaN-based quantum well LEDs operating in the near-UV, blue, and blue-green spectral ranges, the external quantum efficiency of AlGaN quantum well LEDs operating at 280 nm is still below 10%. The external quantum efficiency is further reduced drastically for devices emitting at 240 nm, or shorter wavelengths. Optically pumped AlGaN quantum well lasers that can operate in the UV-B (280-315 nm) and UV-C (100-280 nm) bands have been demonstrated, and the threshold power is generally in the range of 100 kW/cm^2. However, there has been no demonstration of electrically injected semiconductor edge-emitting lasers operating in the UV-B and C bands. The challenges for realizing efficient LEDs and electrically injected lasers in the deep UV band include the presence of extensive dislocations and defects and the extremely poor *p*-type conduction in conventional AlGaN planar structures.

In this context, significant progress has been made in the epitaxy and characterization of AlGaN nanowire heterostructures. Nearly dislocation-free AlGaN nanowire arrays can be grown directly on low-cost, large-area Si substrate. The authors have further discovered, both experimentally and theoretically, that Mg-dopant incorporation is significantly enhanced in nearly defect-free III-nitride nanowire structures compared to their bulk counterparts, thereby leading to very efficient *p*-type conduction in wide bandgap Al-rich AlGaN that was not previously possible. The free hole concentration in AlN nanowires is measured to be ~ 10^{17} cm^{-3}, which is a few orders of magnitude higher compared to the previously reported AlN epilayers. Moreover, the authors have demonstrated the first AlN nanowire LED devices, which exhibit a turn on voltage of 6 V, only limited by the energy bandgap of AlN. More recently, the authors have demonstrated, for the first time, electrically pumped lasers operating in the UV-B band.

In this project, we propose to investigate the epitaxy, and structural and optical properties of Al-rich AlGaN nanowire heterostructures using the technique of selective area epitaxy, which can lead to a precise control of the size and shape of nanowires. We request a deep UV photoluminescence (PL) system for the optical characterization of AlGaN nanowire heterostructures, including the emission spectrum, quantum efficiency, and their dependence on temperature and growth and design parameters. The system includes pump lasers, high resolution spectrometer, deep UV CCD, cryostat, mirrors, lenses, and other related optical components. Such a system is essentially required for the research and development. We propose to demonstrate high efficiency AlGaN nanowire LEDs and electrically pumped AlGaN nanowire lasers operating in the UV-C band.

2. Summary of the Most Important Results

2.1. Deep UV Photoluminescence System

The deep UV optical characterization system includes UV lasers to optically excite the AlGaN samples, a spectrometer and suitable detectors to spectrally resolve and detect the emitted light, a cryostat for such measurements at cryogenic temperatures, and various deep UV-compatible optical and electrical components. The complete system is shown in Figure 1 below.

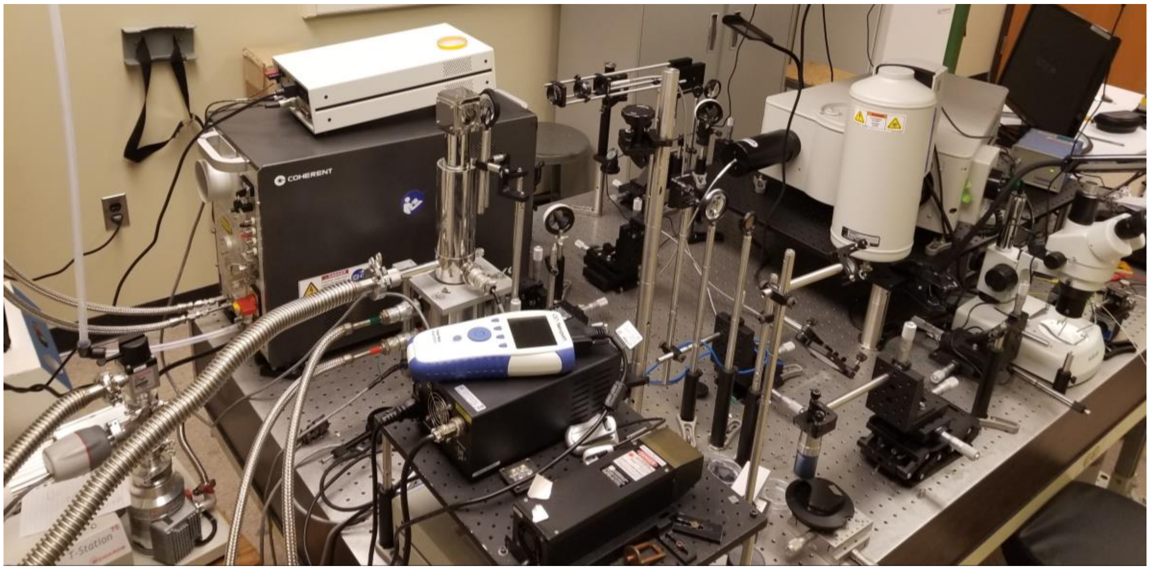

Figure 1: The deep ultraviolet photoluminescence measurement system.

2.2. Optical characterization of AlGaN nanowire heterostructures

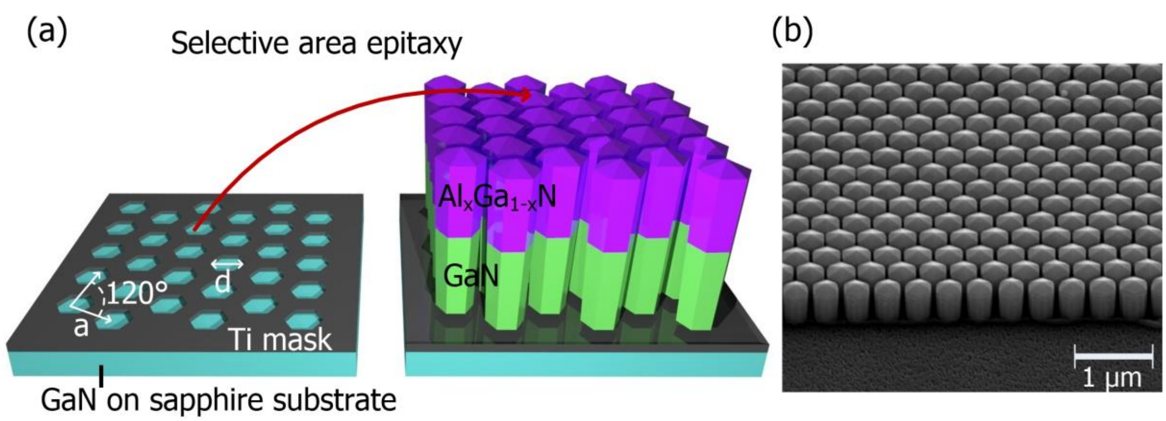

Figure 2. (a) Left: Nanoscale aperture arrays defined by e-beam lithography on a 10 nm thick Ti mask on a c-plane GaN-on-sapphire substrate. Right: Schematic of the selective area epitaxy of GaN/AlxGa1-xN nanowires on the patterned substrate. (b) An SEM image of GaN/AlxGa1-xN nanowire arrays grown by selective area epitaxy.

AlGaN nanowire arrays with Al incorporation controllably varied across nearly the entire compositional range are grown by plasma-assisted molecular beam epitaxy. Illustrated in Figure 2(a) is the selective area epitaxy of $Al_xGa_{1-x}N$ nanowire arrays on a GaN nanowire template formed on c-plane GaN-on-sapphire substrate. A thin Ti layer (~10 nm) was employed as the growth mask for the selective area epitaxy of GaN nanowires. Prior to the growth of nanowires, nitridation of the Ti mask was performed at 400ºC to prevent crack and degradation during the growth process. $Al_xGa_{1-x}N$ nanowires were grown with substrate temperatures and a nitrogen flow rate in the range of 935°C to 1025°C and 0.3 sccm to 0.55 sccm, respectively. Ga and Al beam fluxes were varied in the range of $\sim 10^{-7}$ to $\sim 4.2\times10^{-7}$ Torr and $\sim 10^{-8}$ to $\sim 7.1\times10^{-8}$ Torr, respectively to tune the alloy composition and emission wavelength. Shown in Figure 2(b) is a scanning electron microscope (SEM) image of GaN/$Al_xGa_{1-x}N$ nanowire arrays, which exhibit controlled size and spacing and well-defined hexagonal morphology, with a very high degree of uniformity. Detailed scanning transmission electron microscopy studies further confirm that an Al-rich AlGaN shell structure is spontaneously formed surrounding the nanowires, which can suppress nonradiative surface recombination.

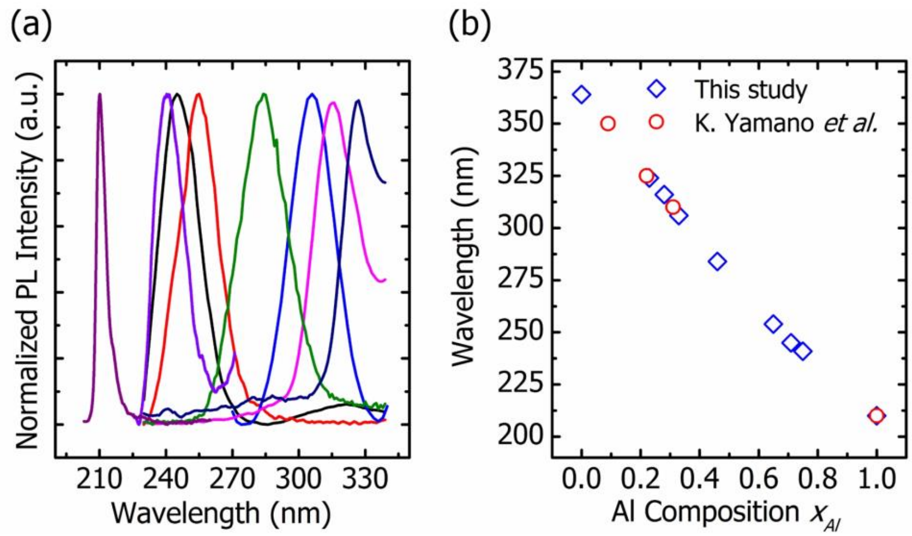

Figure 3. (a) Normalized room-temperature photoluminescence (PL) spectra of $Al_xGa_{1-x}N$ nanowire arrays with Al compositions tuned from ~20% to 100%. (b) Plot of emission wavelength vs. Al composition for AlGaN nanowires demonstrated in this work (blue diamond) and reported previously (red circle) by selective area epitaxy.

Photoluminescence (PL) spectra of these AlxGa1-xN nanowire arrays were measured at room-temperature. The nanowire sample was excited using a 193 nm ArF excimer laser, and the PL emission was collected and spectrally resolved by a high-resolution spectrometer and detected by a liquid nitrogen cooled charge coupled device detector. Illustrated in Figure 3(a), strong PL emission from 210 nm to 327 nm was measured from $Al_xGa_{1-x}N$ nanowires grown with different Al compositions. Illustrated in Figure 3(b) are variations of the PL emission wavelength vs. Al composition. It is seen that $Al_xGa_{1-x}N$ nanowires with Al concentration varying across nearly the entire compositional range, i.e. from x = 0 to 1 can be readily achieved by selective area epitaxy. For comparison, previous studies on selective area epitaxy of $Al_xGa_{1-x}N$ nanowires were largely limited to Al composition below 40%.

The realization of high quality Al-rich $Al_xGa_{1-x}N$ nanowire arrays by selective area epitaxy provides a distinct opportunity to demonstrate high efficiency nanowire photonic crystal light emitters including LEDs and laser diodes operating in UV-C band, which are currently being investigated.